Advanced Accelerator Applications
Transmutation Science Group

Conceptual Design of a Corrosion Probe for Use in Molten Lead-Bismuth Eutectic

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LA-UR-03-0058

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Introduction

In analogy with the science of aqueous corrosion and its real-time measuring techniques, a corrosion probe for a molten lead-bismuth eutectic (LBE) system would measure in real-time the dielectric properties of the protective oxides. From these data we can obtain information such as oxide thickness, which is proportional to corrosion rate. Laboratory studies on candidate materials are in progress to determine the relation between the dielectric properties of the protective oxide layers, which are typically semiconductors, and the metallographic characterization of those oxides, in other words, to understand the kinetics of metal corrosion in LBE. It is anticipated that the DELTA Loop will figure prominently in the determination of the relation between the properties of the oxide layers and metal corrosion rates by means of performing long exposures under controlled oxygen, flow, and temperature. Furthermore, the DELTA Loop could become an ideal laboratory for the kinetics studies provided that corrosion probes can be successfully introduced.

In the laboratory studies, metal samples are partially immersed in LBE with air or a gas above the LBE, and the electrical connection to the sample is made above the LBE level. A fully immersed LBE corrosion probe, suitable for use in the DELTA Loop, must have the following characteristics:
   ❖ electrically isolated from the Loop piping in order to measure the dielectric properties of the oxide surface
   ❖ the insulator and the metal-to-insulator joint must withstand the pressure, fluid forces, temperature, and the corrosive effects of the LBE.

Commercial ceramic-to-metal joints are typically made by first metalizing the surface and then brazing with a variety of brazing alloys that in general are not LBE resistant. [1] Direct brazing is also performed without first metalizing the ceramic, and use alloys of Ti together with Au, Cu, Ag, Pb, or In.[2] The high solubility of the braze alloys used in either technique prevents long term exposure to LBE and introduces unacceptable contamination of the LBE. Another issue is the situation that the braze alloy is in perfect electrical contact with the sample as well as being in contact with the LBE at the braze line. Thus, the electrical response of the braze alloy to the LBE is presented in parallel to that of the sample and interferes with the measurement, for example, by shorting out the signal. Alternate methods of making the insulated joints are considered in this design study.

Glass sealing techniques developed for the APT aqueous corrosion experiments at A6 can be applied to LBE. Glass is a suitable sealing material because it is resistant to LBE and glass-to-metal joints are designed to withstand high temperatures and temperature swings. However, it may be that the glass seals cannot support the velocity pressures and vibrations applied to the corrosion probe by the flowing fluid. Designs with low surface area and streamlined profiles can likely overcome these difficulties, and glass sealing

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should be investigated. It has been our experience in APT (measuring corrosion rates in water) that probes fabricated with glass seals failed more rapidly (weeks) as compared to probes fabricated with tapered compression joints (months or greater).

Inertial welding between the probe material and ceramic creates a strong, leak-free joint. The development period was too long for this promising method to be considered for the APT experiments, but is a candidate for LBE corrosion probe development.

Mechanical metal-to-ceramic joints

Tantalum is a suitable gasket material, if not the only one, for LBE probes because it has a low corrosion rate in LBE; its electrical response can be characterized and is expected to be stable.[a] And it is softer than either the ceramic or the candidate materials for LBE service and should perform well as a gasket by filling cracks and voids in the sealing surfaces. Several types of joints are considered, each using tantalum as the gasket material. A mechanical joint made without a gasket was used in APT aqueous corrosion experiments and is discussed.

Beyond the requirement for sealing, the tantalum is promising for producing crevice-free joints. Crevices[b] introduce uncertainty because oxygen concentration and wetting will be different in these regions. The crevice, to be problematical, must be small enough so that the LBE chemistry is locally determined and not affected by oxygen control or circulation in the main stream. In the glass-sealed joint (and the inertially welded joint), no crevice exists at the transition. While it is interesting to understand what is occurring in the crevices, the effects of the crevices will bias the interpretation of the corrosion of the probe surface in the flowing stream. For example, a low resistance path could be present in the crevice due to loss of the protective oxide and wetting of the metal----thus shorting out the probe response. The effects of crevices can be studied in laboratory experiments or in the DELTA loop using probes with deliberately introduced crevices. To obtain a crevice-free joint, sealing must occur at the first contact line between the probe material and the tantalum gasket.

We present three distinct sealing concepts and consider the application of these in two distinct locations in the DELTA Loop. Further considerations for LBE corrosion probes are introduced along with each proposed concept.

Corrosion probe located in the O$_2$ sensor housing

The first two sealing concepts using tantalum are illustrated in Figures 1 and 2 and utilize the existing O$_2$ sensor components and housing developed by Tim Darling for the DELTA Loop. Installing a corrosion probe in an O$_2$ housing is motivated by the possibility of adapting the available yttria-stabilized zirconia cone as the insulator for the probe.

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[a] Investigation of tantalum by itself in LBE is required before incorporating it into a corrosion probe.
[b] Crevice corrosion is a well know phenomenon in aqueous corrosion.

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VCR-style seal. In the first case, the tantalum seal is patterned after the highly successful Cajon VCR design where a polished metal seal is indented by opposing polished circular ridges that themselves have a hemispherical cross-section. When installed without relative rotation between the opposing components, it provides unrivaled performance for consistent, leak-free, seals from high vacuum to high pressure. They have been used in APT experiments with frequent temperature swings from liquid nitrogen immersion to 200 ^{o}C. In the corrosion probe application of Fig.1, a polished tantalum gasket is indented by the polished nose of the zirconia cone that has been cut off from its original shape shown in Fig. 2. The face of the HT-9, T91, or 316L SS corrosion probe indents the opposing side of the tantalum gasket, and the large force (at least 2000 lbs) necessary to indent the seal is provided by the central screw of Alloy 718 that maintains sufficient strength over the anticipated LBE temperature range in the DELTA Loop. Differential thermal expansion temperature coefficients are handled with Inconel X-750 Belleville springs that are effective at all temperatures considered.

In the VCR-style seal, the angle between the corrosion probe and the tantalum seal at the line of contact exposed to the LBE is approximately 90°, and therefore no crevice is expected.[c]

Because the zirconia is not in pure compression, it is not immediately known whether the rounded nose will withstand the forces without cracking. As assistance, the radial component of force is compressive on the zirconia as the seal deforms. An assembly technique, useful for all the tantalum applications, is to make the seal at the elevated temperature of 200 ^{o}C where the yield strength of the tantalum falls by a factor of three to about 66 MPa. [3]

Preliminary prototyping steps are to measure the force necessary to moderately deform tantalum by opposing VCR noses. The second step is to deform tantalum with a modified zirconia insulator at up to the same load and to examine the nose for damage. Helium leak checking of a prototype seal after thermal cycles is a further development step.

The prospects for this design hinge on the ability of the Alloy 718 screw to deliver sufficient force to make the seal; at 80% of its yield stress, it can pull 2500 lbs which may not be enough. If that is the case, it may still be possible to form the seal with a press at higher axial load and then maintain the seal with the 2500 lbs. This technique is discussed more seriously for the later designs where good alignment is more likely to be maintained as the press is released against the screw with Belleville spring force applied.

Tapered joint. The second tantalum sealing concept is illustrated in Fig 2. It is patterned after 6061-T6 corrosion probes developed for the APT aqueous corrosion program. The

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[c] VCR seals exhibit no virtual leaks in high vacuum situations, another way to express the expectation that the VCR-style seal will have no crevice.

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6061-T6 probe could not use the glass sealing methods without melting. Therefore the aluminum probes were pressed onto a 3° tapered alumina cones. No gasket was used, and a vacuum tight seal was obtained with polished surfaces and boron nitride lubricant to prevent galling. No characteristic crevice signal was observed in the test probe nor in the several probes operated in the high intensity beam at A6.

A design condition for the tapered joint is that differential expansions between the corrosion probe wall and the ceramic not exceed the elastic deformation of the probe wall at a stress that is near the yield point. HT-9 meets the condition while T91 and 316L SS do not:

> The 400 °C HT-9 linear thermal expansion $\Delta L/L$ is 4.44 x 10^{-3}. [4]
> The 400 °C Y PSZ linear thermal expansion $\Delta L/L$ is about 2.7 x 10^{-3}. [5]
> The differential linear thermal expansion $\Delta L/L$ is then 1.7 x 10^{-3}.
> The HT-9 yield stress at 400 °C is 550 MPa. [4]
> The HT-9 modulus of elasticity at 400 °C is 1.72 x 10^{5} MPa. [4]
> HT-9 can therefore store an elastic strain of 0.0032 at 400 °C.
> Hence the differential thermal expansion uses up 54% of the elastic strain.

For the tantalum to be a useful seal, it must be deformed, which is only marginally possible with the dimensions given in Fig 2. HT-9, at its 200-300°C yield strength of 600 MPa, can produce a maximum compressive stress on the tantalum of

$$2 \, \sigma_y \, t/d = 70 \, MPa \qquad (1)$$

which is near its yield point at 200-300°C. Assembly at the elevated temperature takes advantage the high temperature strength of the HT-9 and the reduced tantalum strength. Upon cooling, the HT-9 yields additionally against the ceramic, but since the tantalum strengthens, the tantalum undergoes no further yielding. The HT-9 wall thickness t is limited by the restriction of the existing zirconia seat (Fig. 1). Although thicker HT-9 walls would load the tantalum more, it would take higher axial force for assembly which could break the zirconia cone. The frictionless axial force is

$$2\pi \, \sigma_y \, t \, l \, tan(5.7°/2) = 132 \, lbs. \qquad (2)$$

The length of metal in contact with the insulator is l. Measured axial forces for the APT 6061-T6 and Alloy 625 probes were about 3 times the frictionless force, corresponding to a friction coefficient of 3, even with boron nitride lubricant. So the pressing force is expected to be approximately 400 lbs.

At the 200 °C yield point, the tantalum cannot form a metal-to-metal seal of quality comparable to a VCR seal. It may flow at high spots where the local stress is much higher, but that is the extent of the sealing action. There is not enough pressure available

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to extrude tantalum into the cracks and scratches on the surfaces. It can be said that if the tantalum is smooth and is well-dimensioned, it may help as a gasket because it is much softer than the other materials.

Since the angle between the probe material and the ceramic is approximately 0°, it is speculated the tantalum could improve the probability of achieving a crevice-free seal around the entire probe circumference. Or the tantalum might tear or gall, particularly in such thin gaskets, and certainly fabrication development is needed.[d]
As previously stated, the joints developed for APT seal directly between the probe wall and a 3° tapered Al_2O_3 section. Since these successful joints were applied to aluminum 6061-T6 and Alloy 625 probes without gaskets, there is confidence that the joint would function without a tantalum gasket.

The prospects for the tapered seal are good since the idea has been previously used successfully at A6 in a harsh environment. It is our opinion that the tapered seal promises to deliver the most robust free standing probe (free standing vs. "pipe section" described below). The greatest detractor is the inability of this design to accommodate 316L. See the Table 1 summary of corrosion probe options.

Corrosion probe installed at the removable pipe section
Three distinct sealing methods are examined for installation of the corrosion probe assembly in one of the 12 inch long removable pipe sections of the DELTA Loop or in the Test Section if space permits.

Garlock seals in axial configuration. An entirely different sealing approach (Fig. 3) shows tantalum as the sealing surface coated on Garlock spring-loaded seals pressing on ceramic disks. Seen in cross-section, the corrosion probe is an insulated 2 inch diameter pipe section. Eight Belleville-loaded bolts compress the Garlock seal on each flange of the pipe section. The distance between the 6 inch diameter 316L SS flanges is 12 inches.

As with the VCR seals, the commercial Garlock seals have a non-zero contact angle and can be expected to seal the LBE at first contact, resulting in crevice-free joints.[e] To the extent that corrosion in LBE is a combined corrosion/ erosion effect, this version is ideal with uniform velocity over the entire surface of the corrosion probe.

A secure secondary LBE containment is required for all insulated joint concepts. The secondary is a complication in Fig. 3 because it adopts the form of a larger diameter pipe outside the ceramic-to-Garlock seals. It is necessary to decouple the axial loading of the large outer flanges from applying excessive force to the Garlock-to-ceramic seals. These forces arise due to differential expansions, construction tolerances, variations of Grafoil[f]

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[d] Tantalum has been extruded into soda can shapes for earlier projects.
[e] Certain commercial aqueous corrosion probes are sealed with Viton O-rings and do not have crevice corrosion, demonstrating that the similar Garlock seal will not either.
[f] Graphite gaskets are used in all of the DELTA Loop connections.

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gasket compressions on the outer flanges, and alignment. In Fig. 3, this decoupling is achieved through the flexible section machined into the upper T91 flange.

The ceramic insulators do not contact the flanges except for alignment purposes. Depending on selection, the Garlock seals require a loading of 2800 lbs per inch. During the 6061-T6 aqueous probe pressing operation, alumina was loaded without difficulty to one half of this value at a distance of 1/8 inch from the edge of alumina.

T91 is used for the flanges of the secondary containment. The high-temperature T91 strength permits a reduction in size of these flanges as compared with their equivalent in 316L SS. In general, it is thought that experience with T91 also will be useful. There will be relative radial motion of the dissimilar 316L and T91 flanges during temperature cycles that must be absorbed by the Grafoil gasket. Axial motion is absorbed by Belleville disc springs designed for heavily bolted flanges. In flange applications where the gasket is compressed under the bolts, the Bellevilles replace the elasticity that is present in ASA flanges due to bending.

The axial configuration is the most robust of all the designs for handling the velocity pressure of the flowing LBE, contrasted with all the others that are free standing and transverse to the flow.

Garlock seal in transverse configuration. Figure 4 shows a different application of the Garlock seal where the probe intercepts flow from the side, and the LBE flow pipe fits between flanged joints with the same 12 spacing as Fig. 3. The secondary containment is simpler in the transverse configuration than for the axial one.

Regions of low flow or stagnant flow are present in this design. This version is seen as a preliminary to the axial configuration because it is easier to change probes in the developmental stage. The ratio of tantalum surface area to probe surface area exposed to LBE is highest for this configuration, but the significance of this fact is not known at this time.[g] HT-9 can be considered for this probe concept if available from Timken in thick plate and large diameter bar stock.

Tapered fit in transverse configuration. Figure 5 explores the tapered fit without the constraints of the existing zirconia component (Figure 2). It is seen that the probe is in a full flow region. It is constrained by a bolt from escaping into the fluid and uses the preferred 3° taper. The upper insulator seal is among the best presented in this report. The aspect ratio of the insulator is adjusted to accept very high axial loads during the pressing of the upper and lower joints. A most important improvement calls for a ceramic with a larger thermal expansion coefficient, and there is frequent mention of zirconia in the range of 10.5×10^{-6} /^{o}C [6] [7], which is excellent for use with HT-9 and adequate for T91. With a press fit on such a ceramic, differential expansion uses up only 20% of the

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[g] The tantalum capacitance needs to be measured and its stability under oxygen control conditions in LBE needs to be determined.

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elastic strain of HT-9 and, less favorably, 38% of the elastic strain of T91 at stresses near the yield stresses. (A 316L SS probe would loosen.)

A second design condition is that stress relaxation (related to creep) not loosen the joint. For T91, the creep rate is 1 x10^{-7} per hour at 270 MPa and 755K. [8] For 1000 hours at temperature, the creep strain is 7% of the elastic strain which is quite acceptable. It might be assumed that only 7% of the elastic stress will relax at zero strain under the same conditions. (Note also that the stress quoted is 70% of the yield stress in order to obtain the low creep rate.) The high temperatures materials are thus able to maintain high compression on the seal and the sealing surfaces during temperature cycles.

The optimum condition for the metals before sealing is the annealed state. Work hardening is minimized in this way which also with minimizes stress relaxation.

The effective wall thickness *t* in Equations 1 and 2 becomes large for the upper seal, and the limitation on loading becomes the tensile forces in the top edge of the ceramic just outside the pressing foot. As the ceramic is forced into the T91 flange, compressive components are generated in the ceramic that tend to offset the tensile forces generated by the pressure foot.[h], and it is imagined that pressures could be generated in the tantalum that approach workable extrusion pressures (typically 2-4 times the yield stress). At that point the tantalum is acting to locally relieve high stresses in the ceramic and at the same time is making a quality seal. This feature of the tantalum becomes important if the angle of the seat is not exactly the same as the insulator.

The retainer nut is not expected to drive the cone into the flange because a large diameter nut requires high torque for relatively little axial force generated. Its purpose is to align the pressing foot, to start the joint with good alignment[i], and to help maintain alignment as pressing proceeds. The retainer nut is torqued continuously as the pressing proceeds at 200 $^{\circ}$C. The retainer function is there in case the joint tries to loosen under the thermal expansion of the T91, and the web on the top of the nut is sized to apply some elasticity during thermal cycles. Development of the joint parameters is accomplished by examining the tantalum surfaces after assembly to specific axial penetrations. This is best done by cutting away the T91 flange. Requirements for the degree of polish of the surfaces are established in the same way.

The corrosion probe seal is pressed next and is likewise not constrained except for the same tensile forces in top edge of the ceramic under the pressing foot. Hence the probe wall thickness should be the maximum that still permits the probe to press onto the insulator without breaking the insulator on the top edge. In this way large pressures can be generated in the tantalum (at 200 $^{\circ}$C) to extrude it into the cracks and scratches and to form a crevice-free seal at the top edge. Fixturing is possible where the center bolt can be tightened as pressing proceeds. Here again, the bolt is intended to start the pressing with

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[h] This would be a reason to make upper tapered seal first.
[i] The pressure face must be exactly parallel to the thread for there to be any alignment benefit.

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good alignment and to maintain the alignment. It also ensures that the joint stays tight during thermal cycles.

A special application for 316L might be interesting; as 316L loosens on the ceramic during heating, and the seal is maintained by the axial motion provided by the Belleville springs. Then upon cooling, it yields and breaks the protective oxide layers, offering a test of the repair properties of the LBE. A tall Belleville stack would make several thermal cycles available for the experiment.

As expressed earlier, the likelihood of success for this design is the highest of those mentioned: It has the most robust design of the free standing probes; the joint has the potential to form a helium-leak-tight seal; if necessary, it could work without tantalum (and has); and may even simplify to a version without threaded retainers. The biggest detractor is inability to accommodate 316L.

VCR-style in transverse configuration. Figure 6 looks at the VCR-style tantalum seal without the constraints of the existing zirconia component (Figure 1). The main advantage here is the stability of the ceramic. The forces on the narrowly rounded nose are similar. And again, the main issue is whether the bolt can provide the required force to deform the tantalum seal at 200 $^\circ$C. The length of seal nearly triples, but the bolt area more than quadruples (3/8 bolt). Force that the bolt cannot comfortable supply, could be delivered by the same press that made the upper seal. A VCR sealing gasket is generally changed if the seal is broken. However, a seal that loosens to the point of developing a leak can frequently be re-tightened to regain the seal. The question is whether the forming and sealing action of the press be maintained when its force is released to the lower level provided by the Belleville springs. The answer is likely yes, if the press does not misalign the probe. That issue is determined by the quality of the press and the fixturing. By combining a stable pressing platform on top of the ceramic with a central, coaxial, force on the bottom of the probe, there is a good chance of success.

Additional considerations

1) Probe design is affected by the order of the assembly steps, as to whether pre-oxidation of the probe surface precedes assembly or follows it. Probe designs using tantalum favor pre-oxidation before assembly due to tantalum oxidation at high temperature in air. Even then, loose oxide should be removed before making the seal which will leave some probe surface area that must be re-oxidized before immersion in LBE. Most of the applications require pressure on the probe which will damage the oxide and require repair before immersion in LBE. Thus far, pre-oxidation has been conducted at temperature too high for the tantalum in air, and lower temperature pre-oxidation needs to be investigated before tantalum can be used.

2) Two probes must be installed so one can serve as a reference electrode for the other.

3) The insulator and seals must withstand thermal shock that comes with filling the Loop with LBE from the melt tank. This situation arises when the component is not

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controlled at the same temperature as the incoming fluid. Glass seals might be particularly susceptible.

4) Continuous leak checking for LBE into the secondary containment is important. Electrical level sensors are used for this purpose. Since it is anticipated that inert gas would be flowed into the secondary to prevent oxidation of the components, some additional use of the gas could be made: pressure testing of secondary, sensing helium on the LBE side, or level sensing using a sensitive Baratron (with gas flow off).

Conclusions

Several conceptual design options are presented that can support the fundamental laboratory studies on the kinetics of metal corrosion in LBE. The initial design chosen may not be the final one because the work in LBE is new. The preferred tapered joint does not have universal application to all materials. See the tabular summary in Table 1 of applicability of the several options and some of the perceived limitations of each.

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3. ASM Handbook Volume 2, page 1163.
4. HT-9 Chapter in Materials Handbook.
5. AC Rochester Division, General Motors Corp. Drawing 5619267
6. Engineered Materials Handbook, Volume 4, page 974.
7. Engineered Materials Handbook, Volume 4, page 990.
8. T91 Chapter in Materials Handbook.

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Electrical signal

Fixed nut

Alloy 718 screw

Rotating nut

Belleville spring

Modified
zirconia cone

Existing seat in
the oxygen
sensor housing

Ta gasket

10-32 LHT

HT-9, T91, or 316
corrosion probe
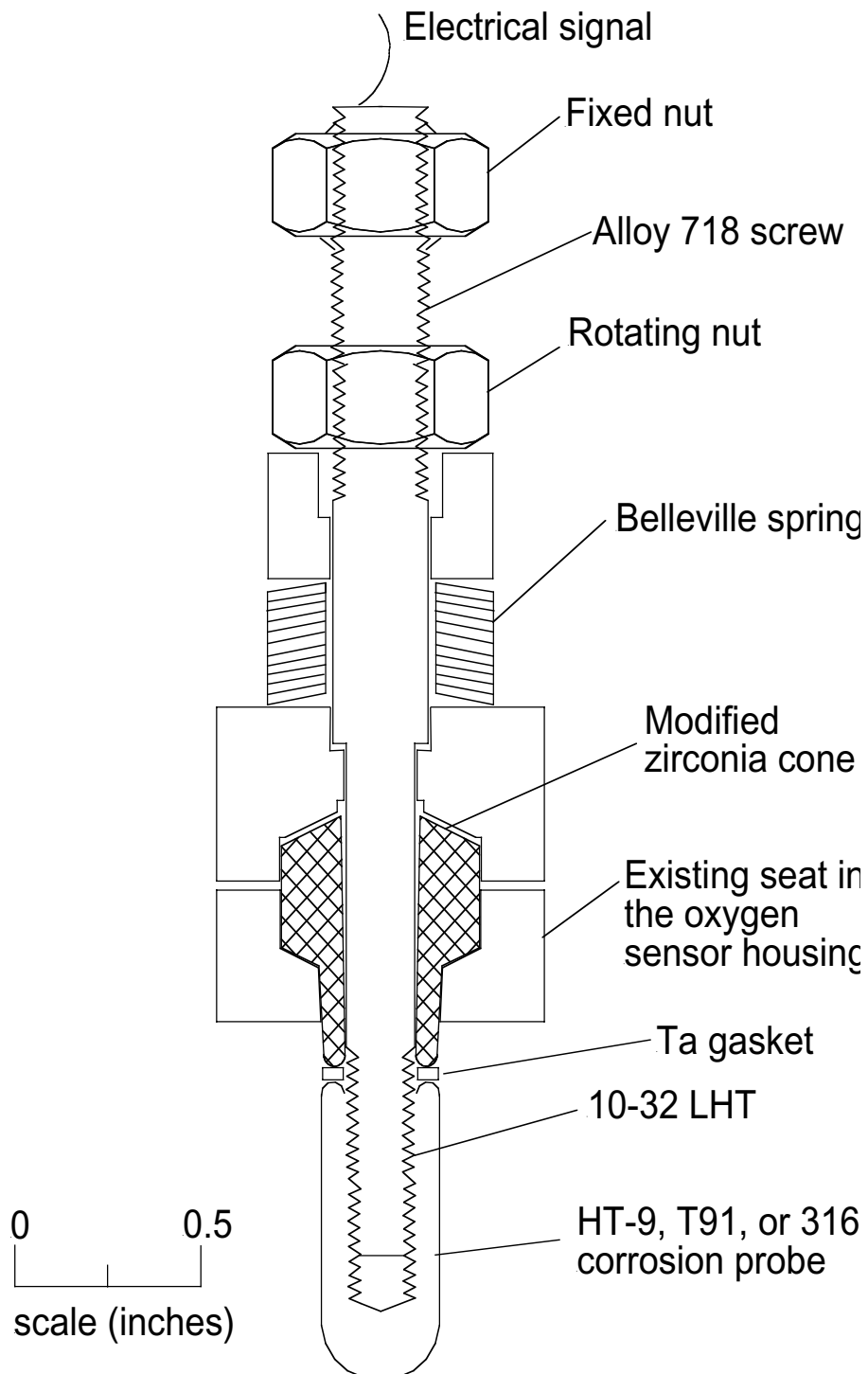
0   0.5

scale (inches)

Figure 1. Cross-section of the VCR-style seal with Ta gasket indented between rounded zirconia nose and corrosion probe. Assembly fits inside O₂ sensor housing.
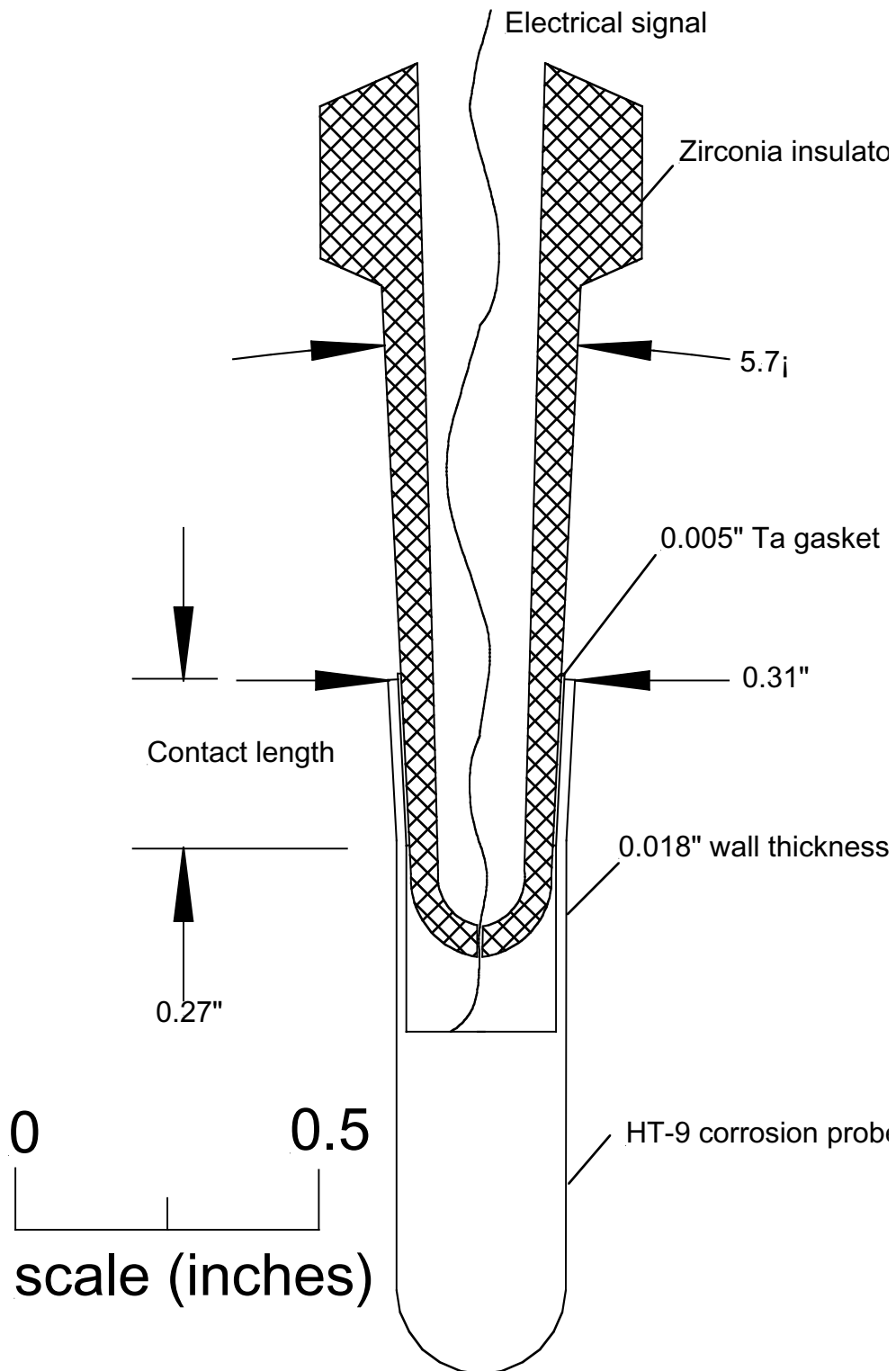
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Figure 2. Cross-section through tapered joint with Ta seal. Suitable for HT-9 only.

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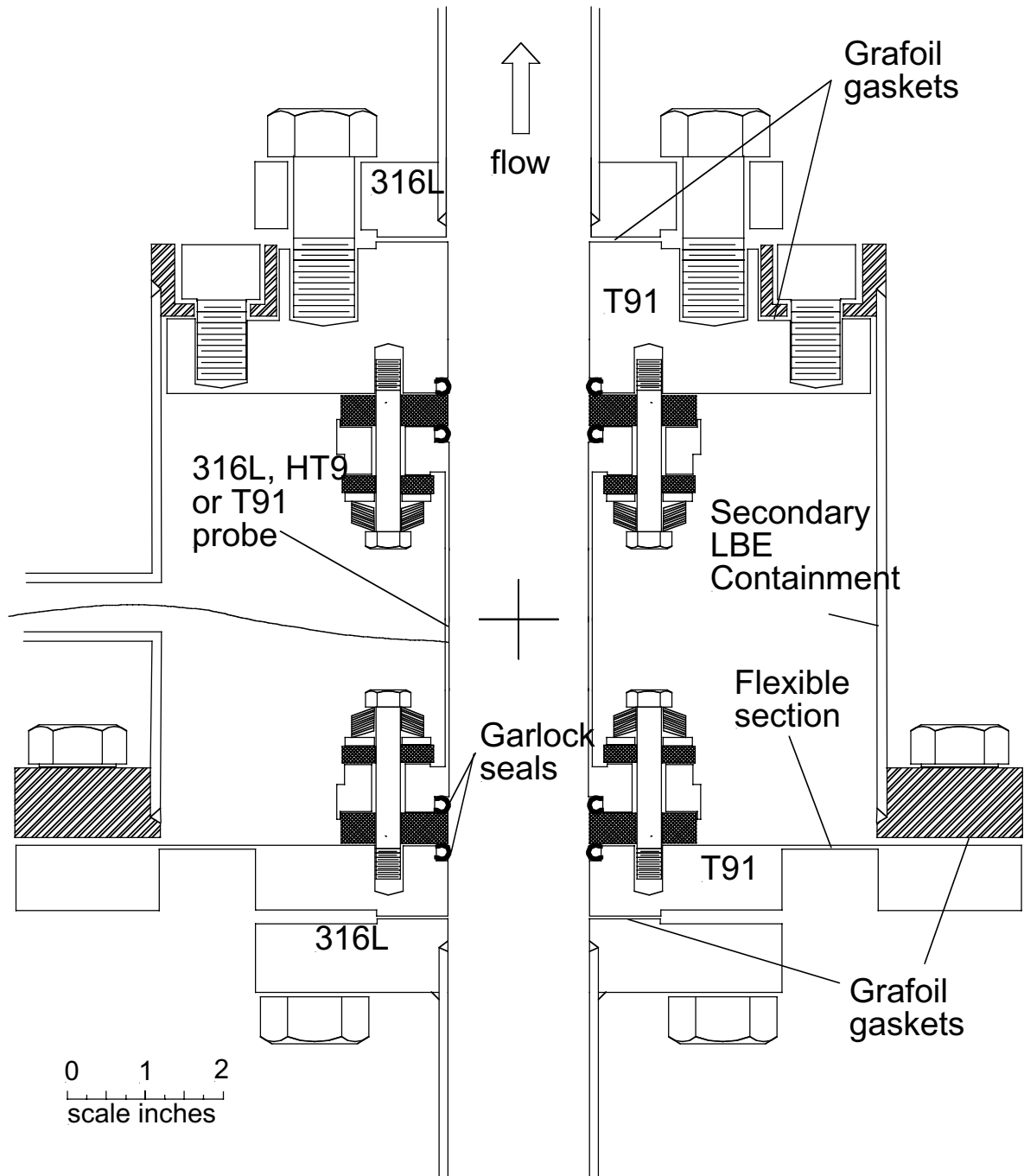for Use in Molten Lead-Bismuth Eutectic
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Figure 3. Cross-section through ceramic-insulated pipe section which comprises the corrosion probe. Ta coated Garlock seals are compressed against the ceramic with Belleville-spring loaded bolts. Spacing between 316L flanges is 12 inches.

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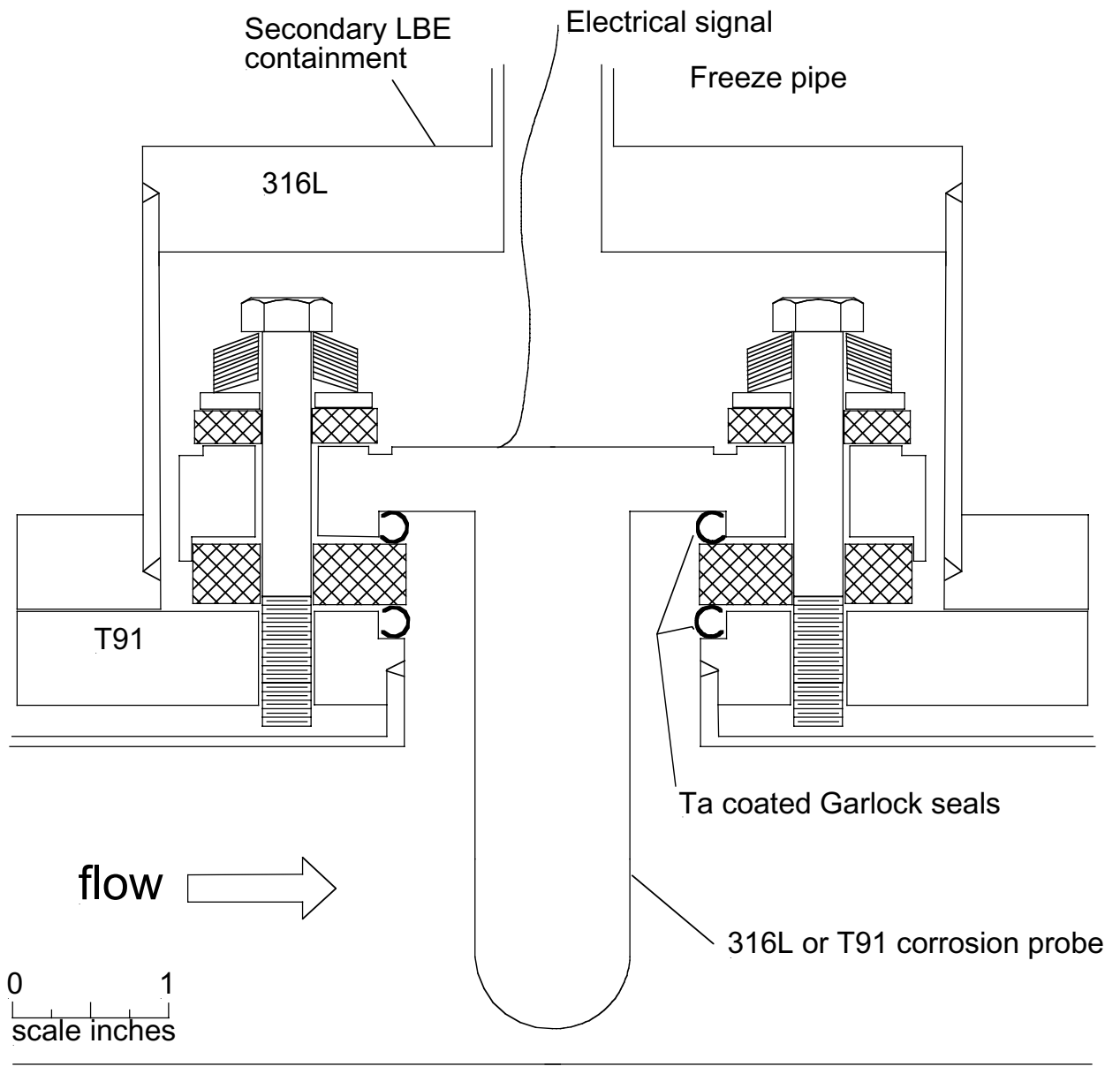for Use in Molten Lead-Bismuth Eutectic
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Figure 4. Variation of the Ta-coated Garlock sealing method on ceramic. Flow pipe fits between flanged joints in the DELTA Loop. Upper regions have low or stagnant flow. HT-9 may not be available in large pieces.
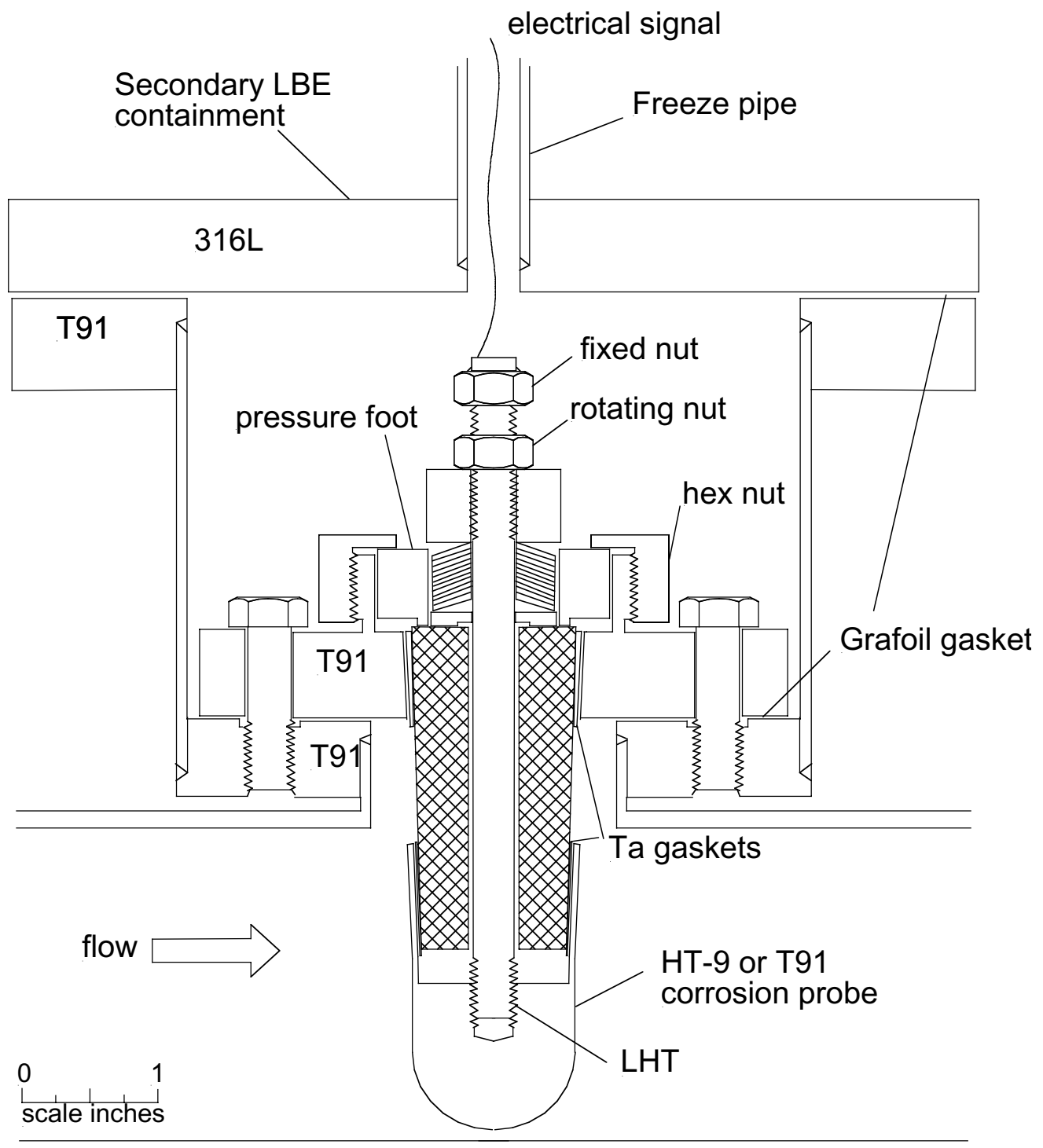
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Figure 5. Cross-section through a Ta-sealed press-fit assembly. Uses 3° taper for upper
and lower joints.

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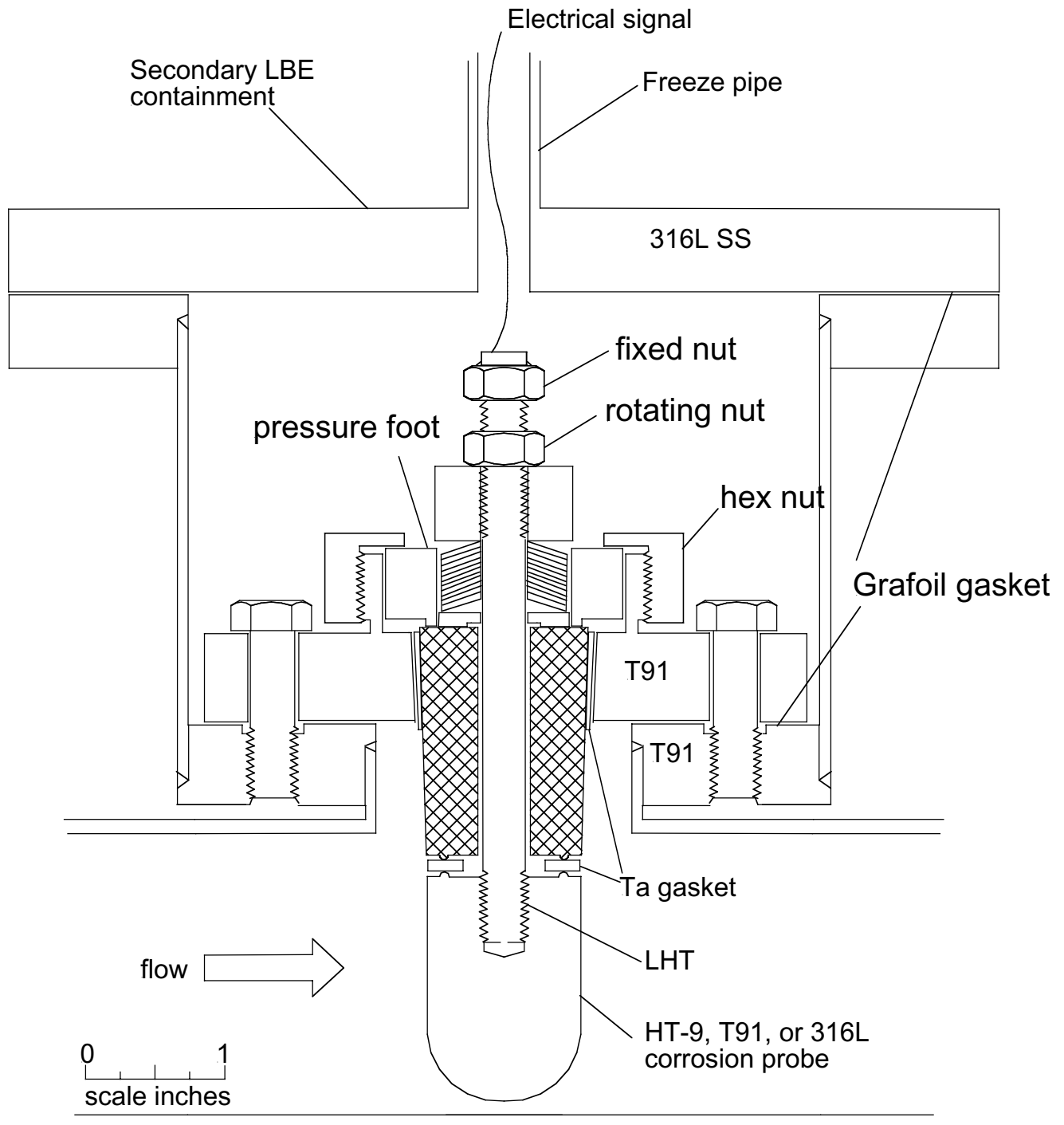for Use in Molten Lead-Bismuth Eutectic
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Figure 6. Cross-section through a VCR-style Ta seal between ceramic and corrosion probe noses. Differential expansions are absorbed by Belleville springs.

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Style	VCR seal on Y PSZ	Taper on Y PSZ	Garlock axial	Garlock transverse	Taper	VCR	Glass sealed	Inertial welded
Figure	1	2	3	4	5	6		
Location	oxygen sensor	oxygen sensor	12 in spool	12 in spool	12 in spool	12 in spool	12 in spool	12 in spool
HT-9	Y	Y	Y	N	Y	Y	Y	?
T91	Y	**N**	Y	Y	Y	Y	Y	?
316L	Y	**N**	Y	Y	**N**	Y	Y	?
Seal Material	Ta	Ta or none	Ta	Ta	Ta or none	Ta	glass	none
Ceramic stress	Comp/Tens	Comp/Tens	Comp	Comp	Comp	Comp/Tens	Comp/Tens	Tens
Resistance to damage from LBE velocity pressure	Poor	OK	Excellent	OK	Excellent	Good	Poor	Excellent
Ratio of exposed Ta surface to probe area	small	smallest	high	highest	smallest	small	none	none
Highest temperature capability		Y			Y		?	?

Table 1. Summary of Corrosion Probe Options